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Cobalt-catalyzed regiodivergent hydroalkenylation of alkenes

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ABSTRACT

The direct hydroalkenylation of alkenes represents the most straightforward method for constructing $C(sp^2)$-$C(sp^3)$ bonds. However, controlling chemo- and regioselectivity between two different alkenes remains a fundamental challenge. Herein, we developed a cobalt-catalyzed system to regioselectively ligate electron-deficient alkenes with electron-rich alkenes. The *in-situ* generated Co^{III}-H activate electron-deficient alkenes through a Markovnikov or *anti*-Markovnikov manner, to form linear or branched alkyl-cobalt species as key intermediate, which can successfully achieve regiodivergent hydroalkenylation of acrylates and analogs. The method demonstrates a broad substrate scope and functional group compatibility. Origin of regioselectivity control by the cobalt catalyst has been elucidated through mechanistic studies and density functional theory (DFT) calculations.

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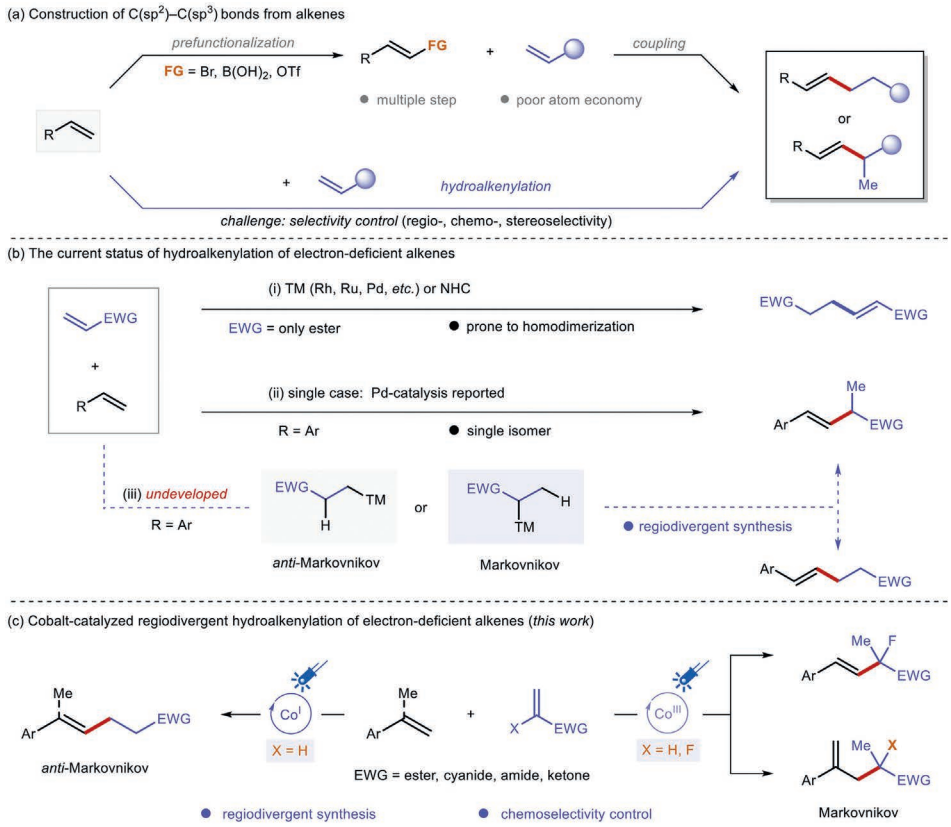
Transition-metal-catalyzed alkene-alkene cross-coupling represents a powerful strategy for the construction of $C(sp^2)$-$C(sp^3)$ bonds [1–6]. This field is currently dominated by two distinct pathways (Scheme 1a). One approach utilizes prefunctionalized alkenes to achieve selectivity control [7–10], albeit with sacrifice of synthetic efficiency due to the additional steps for the installation of functional group to facilitate cross-coupling reaction [11–18]. Alternatively, the direct hydroalkenylation of unfunctionalized alkenes represents an ideal strategy with high atom and step economy [19–22]. However, hydroalkenylation is inherently challenging caused by controlling chemoselectivity (cross-coupling *vs.* homodimerization), regioselectivity (linear *vs.* branched), and stereoselectivity (*Z* configuration *vs. E* configuration), owing to the similar properties of two alkenes [1,3,20–22]. These issues considerably limited its development, and multiple control over selectivity is essential for the efficient preparation of target molecules.

To address these issues, several strategies have been developed, such as Lewis acid catalysis [23–26], transition-metal catalysis [27–33], and NHC catalysis [34,35], which enabled selective hydroalkenylation of specific alkenes. Generally, these systems lead to generate homodimerization products of electron-deficient alkenes (Scheme 1b, i). A significant advance was reported by Gevorgyan and co-workers, who described a visible-light-induced Pd-catalyzed system for Markovnikov-type hydroalkenylation of electron-deficient alkenes [36]. The reaction allows for the preparation of branched cross-addition products between electron-deficient and electron-rich alkenes (Scheme 1b, ii). In principle, both linear and branched products can be obtained by precisely controlling the hydrometallation intermediate derived from one alkene [37,38] and its subsequent reaction with another alkene molecule (Scheme 1b, iii). As a proof of concept, we recently achieved switchable synthesis of adipates and glutarates from two similar acrylates [39]. To extend the scope and utility of this strategy, we now focus on the coupling of alkenes with markedly different electronic properties. Herein, we report cobalt-catalyzed system by modulating the interaction mode between *in situ*-generated Co^{III}-H species and electron-deficient alkenes under visible light irradiation. Both linear and branched adducts can be selectively accessed under slightly different reaction conditions with single regioselectivity (Scheme 1c).

We began our investigation by optimizing the reaction conditions for *anti*-Markovnikov hydroalkenylation using styrene **1a** and methyl acrylate **2a** as model substrates. The combination of $Co(dmgH)_2(DMAP)^nBu$ as the catalyst and catalytic amount of BTMG (2-^{t}Bu-1,1,3,3,-tetramethylguanidine) as the base was identified as the most effective system, affording the *anti*-Markovnikov

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Scheme 1. Background of electron-deficient alkene hydroalkenylation.

product **3a** in 69 % isolated yield as a single *E*–isomer within the limits of detection (Table S1 in Supporting information). Control experiments show that both base and light irradiation are indispensable for the transformation. Even at 150 °C, the reaction cannot proceed without light irradiation.

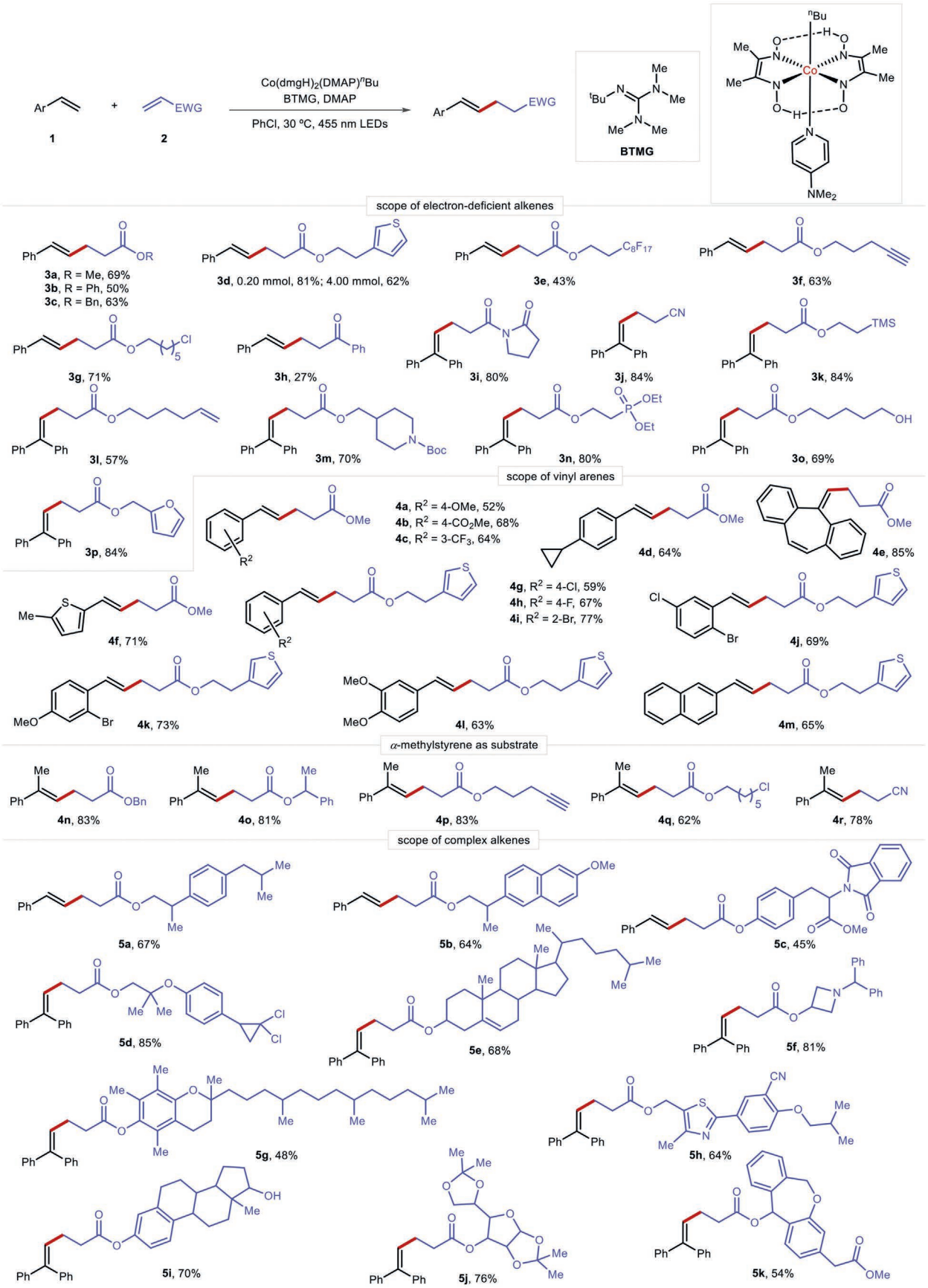
Various electron-deficient alkenes including methyl acrylate (**3a**), phenyl acrylate (**3b**) and benzyl acrylate (**3c**) were found to be suitable substrates (Scheme 2). Scale up the reaction to 4.0 mmol is also successful with the formation of **3d** in 62 % yield. The reaction also showed good compatibility with terminal alkynes (**3f**), and cholorine atom (**3g**), resulting in moderate yields. In addition, acrylketone (**3h**), acrylamide (**3i**) and acrylonitrile (**3j**) can all be readily converted to obtain the *anti*-Markovnikov type products. Moreover, electron-deficient alkenes with various functional groups such as silyl, terminal alkenes, protected piperidines, phosphate esters, free alcohols, furans showed high tolerance in the reaction with 1,1-diphenylethylene, affording **3k-3p** successfully.

Furthermore, styrenes bearing various substituents could be converted to the corresponding hydroalkenylation products **4a-4m**. Additionally, when α-methylstyrenes were used as the substrates, affording **4n-4r** with excellent regioselectivity, the ratio of internal alkenes over terminal alkenes are all greater than 10:1 (see Pages S277–S286 in Supporting information). In addition, structurally complex alkenes as well as drug-like acrylates derivatives (**5a-5k**) were also evaluated, and the *anti*-Markovnikov type cross-addition were successfully carried out, demonstrating high chemoselectivity and functional group tolerance of this protocol.

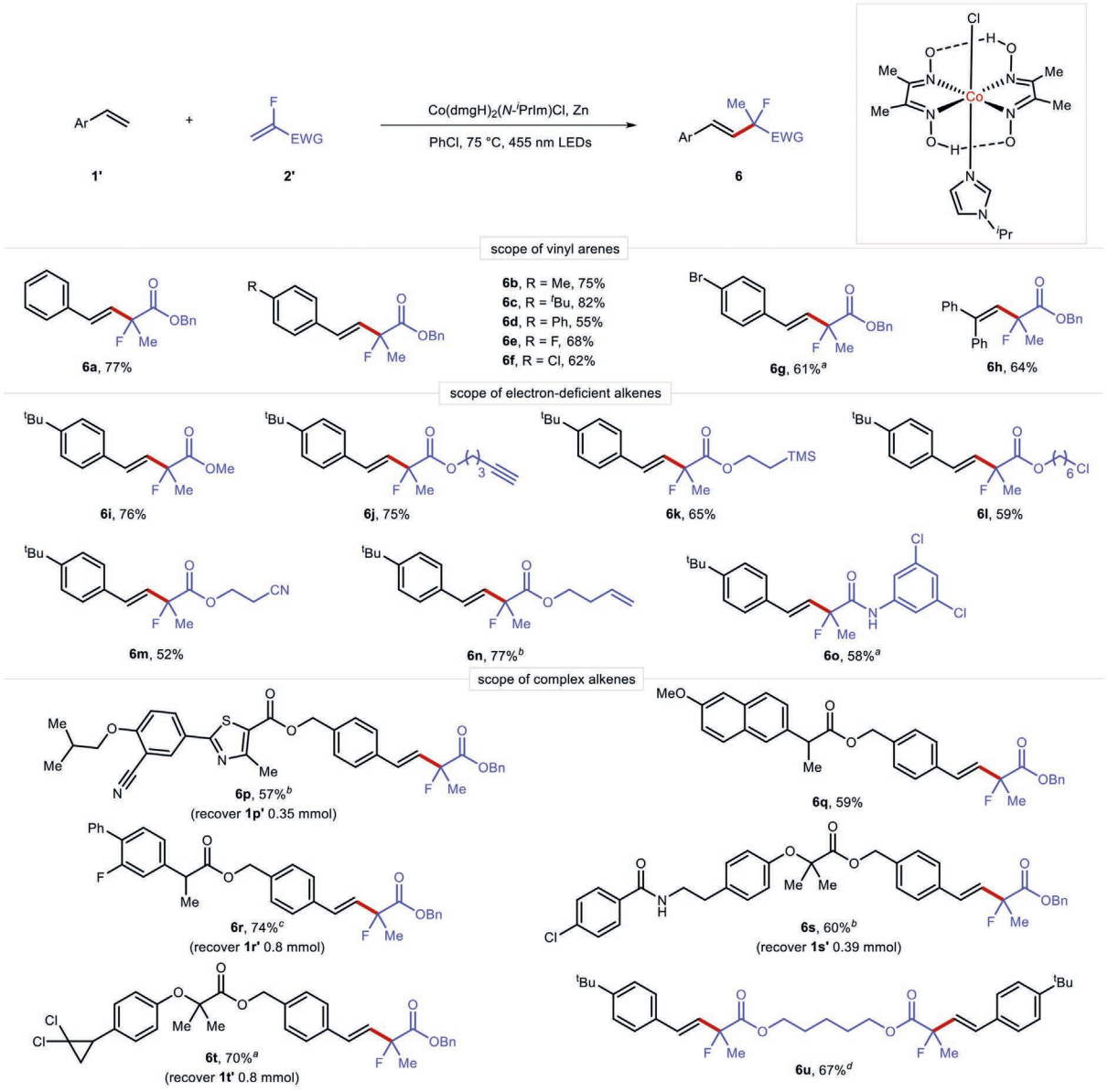
Next, styrene and benzyl acrylate were employed as model substrates to explore the possibility of achieving Markovnikov-selectivity in the hydroalkenylation. After intensive optimization revealed that the use of benzyl 2-fluoroacrylate significantly improved the reaction outcome, affording product **6a** in 77 % yield as a single *E*–isomer within the limits of detection (Table S2 in Supporting information). We believe the fluorine atom enhances the polarization of the C=C double bond compared to benzyl acrylate. The decreased electron density facilitates the metal-hydride-atom-transfer (MHAT) process between the Co-H species and the fluorinated acrylate. Furthermore, the fluorine atom can stabilize the radical intermediate generated through MHAT process, thereby promoting the subsequent C–C bond formation through radical addition (for details, see the mechanism described below). It is noteworthy that the target product **6a** was also obtained in moderate yield when $Co(dmgH)_2(DMAP)^nBu$ was used instead of $Co(dmgH)_2(N\text{-}^i PrIm)Cl$ and Zn.

Styrenes with either electron-donating or electron-withdrawing groups were smoothly converted to the desired Markovnikov-type products **6a-6g** with high regio- and chemoselectivity (Scheme 3). Notably, methyl 2-fluoroacrylate, including sensitive functional groups such as alkynyl, silyl, halogen, cyano, and alkenyl, were all suitable substrates, leading to the formation of **6i-6n** in 52 %–77 % yields. In addition, 2-fluoroacrylamide (**6o**) is also suitable for the cross-addition reaction. Moreover, substrates derived from febuxostat (**6p**), naproxen (**6q**), flurbiprofen (**6r**), bezafibrate (**6s**), and ciprofibrate (**6t**) were all tolerated in the reaction and gave the target products in yields ranging from 57 % to 74 %. When difunctionalized substrates were employed in the reaction, both reacting sites can achieve addition reaction with stryene to form the product **6u**. It is worth noting that although an excess amount of styrene derivatives was required, the reaction proceeded at almost equimolar ratios and allowed for the recovery of unreacted alkenes as shown in **6t**.

Moreover, we assess that the use of α-methylstyrene as feedstock influences the chemoselectivity of the reaction (Scheme 4). Initially, α-methylstyrene and methyl acrylate were employed as model substrates. The reaction afforded the terminal alkene **7a** in 48 % isolated yield, exclusively as the *E*-isomer (within the limits



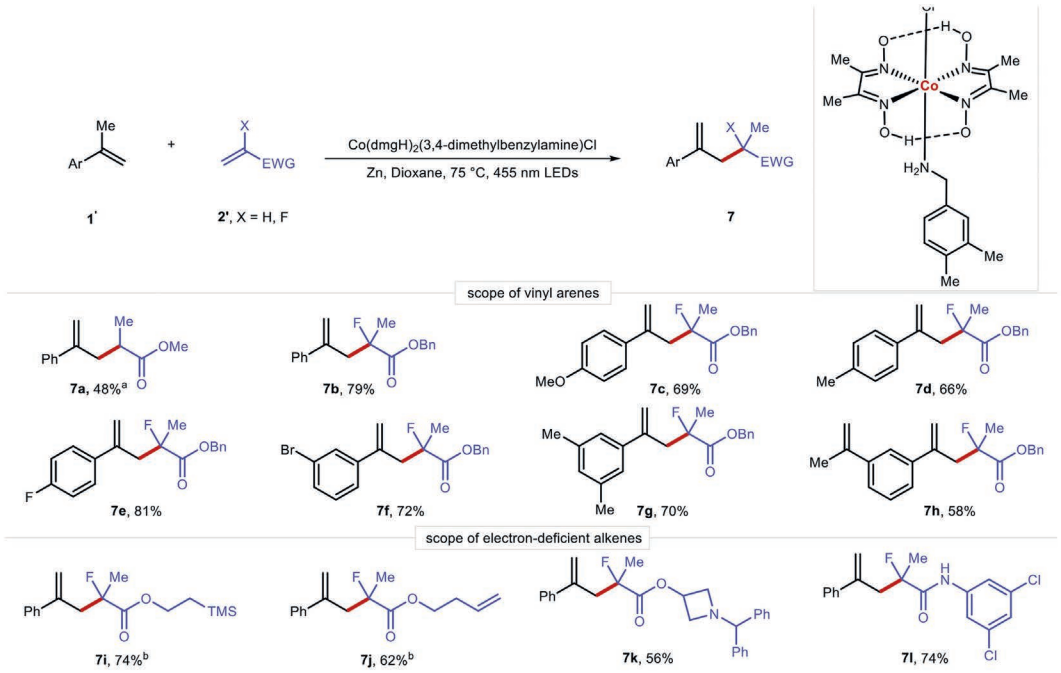
Scheme 2. *anti*-Markovnikov-selective hydroalkenylation of acrylates with styrenes. Standard conditions: 0.20 mmol scale: **1**:**2** = 5:1, $Co(dmgH)_2(DMAP)^nBu$ (20 mol%), BTMG (2-^{t}Bu-1,1,3,3,-tetramethylguanidine) (40 mol%), DMAP (4-*N*,*N*-dimethylaminopyridine) (2.0 equiv.), PhCl (c = 0.2 mol/L) at 30 °C for 24 h under Ar.



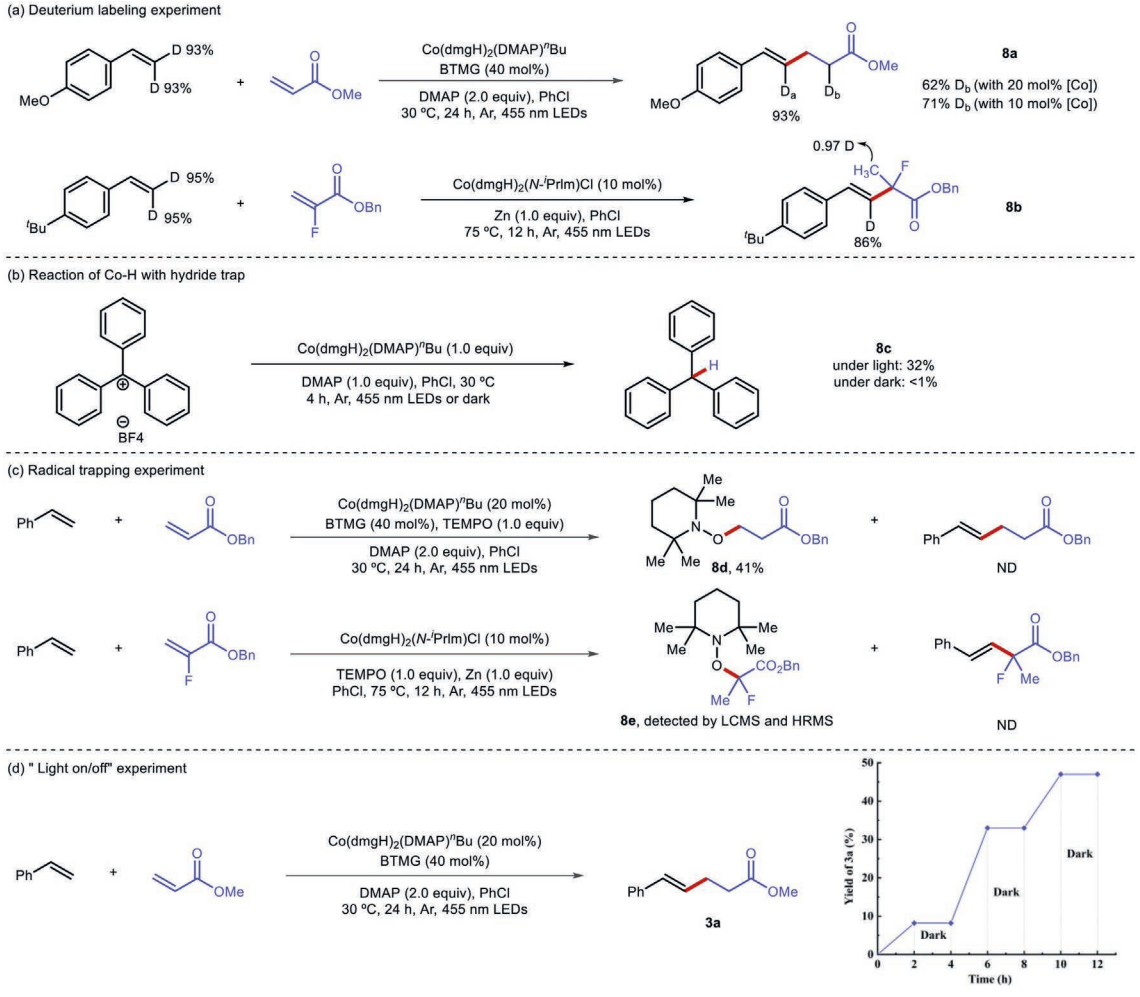
Scheme 3. Markovnikov-selective hydroalkenylation of acrylates with styrenes. Standard conditions: 0.20 mmol scale, **1′**: **2′** = 3:1, Co(dmgH)$_2$(*N*-^{i}PrIm)Cl (10 mol%), Zn (1.0 equiv.), PhCl (c = 0.2 mol/L) at 75 °C for 12 h under Ar. [a] 0.20 mmol scale, **1′**: **2′** = 5: 1, Co(dmgH)$_2$(DMAP)^{n}Bu (20 mol%); [b] 0.10 mmol scale, **1′**: **2′** = 5: 1; [c] 0.20 mmol scale, **1′**: **2′** = 5: 1; [d] 0.10 mmol scale, **1′**: **2′** = 10: 1, Co(dmgH)$_2$(*N*-^{i}PrIm)Cl (20 mol%), Zn (2.0 equiv.). *N-^{i}PrIm = 1-isopropylimidazole.

of detection), with a chemoselectivity of 16:1 for the terminal over the internal alkene (see Page S372 in Supporting information). After further optimization of conditions and substrates, the terminal alkenes products were obtained with excellent chemoselectivity and stereoselectivity (only the *E*-isomer was observed within the limits of detection). Various mono- and di-substituted methylstyrenes (**7b-7g**) could be smoothly converted in 66 %–81 % yields. Especially for diisopropenylbenzene, this protocol also affords the expected product **7h** with high regio- and chemoselectivity. In addition, acrylates and acrylamides containing sensitive functional groups silyl and alkenyl groups could be successfully converted to obtain the products **7i-7l** in good yields with Markovnikov selectivity. Although other long-chain α-substituted styrenes, such as α-propylstyrene, α-butylstyrene, and α-isopropylstyrene, can also serve as coupling partners, they produce *Z*/*E* isomers, resulting in limited synthetic utility.

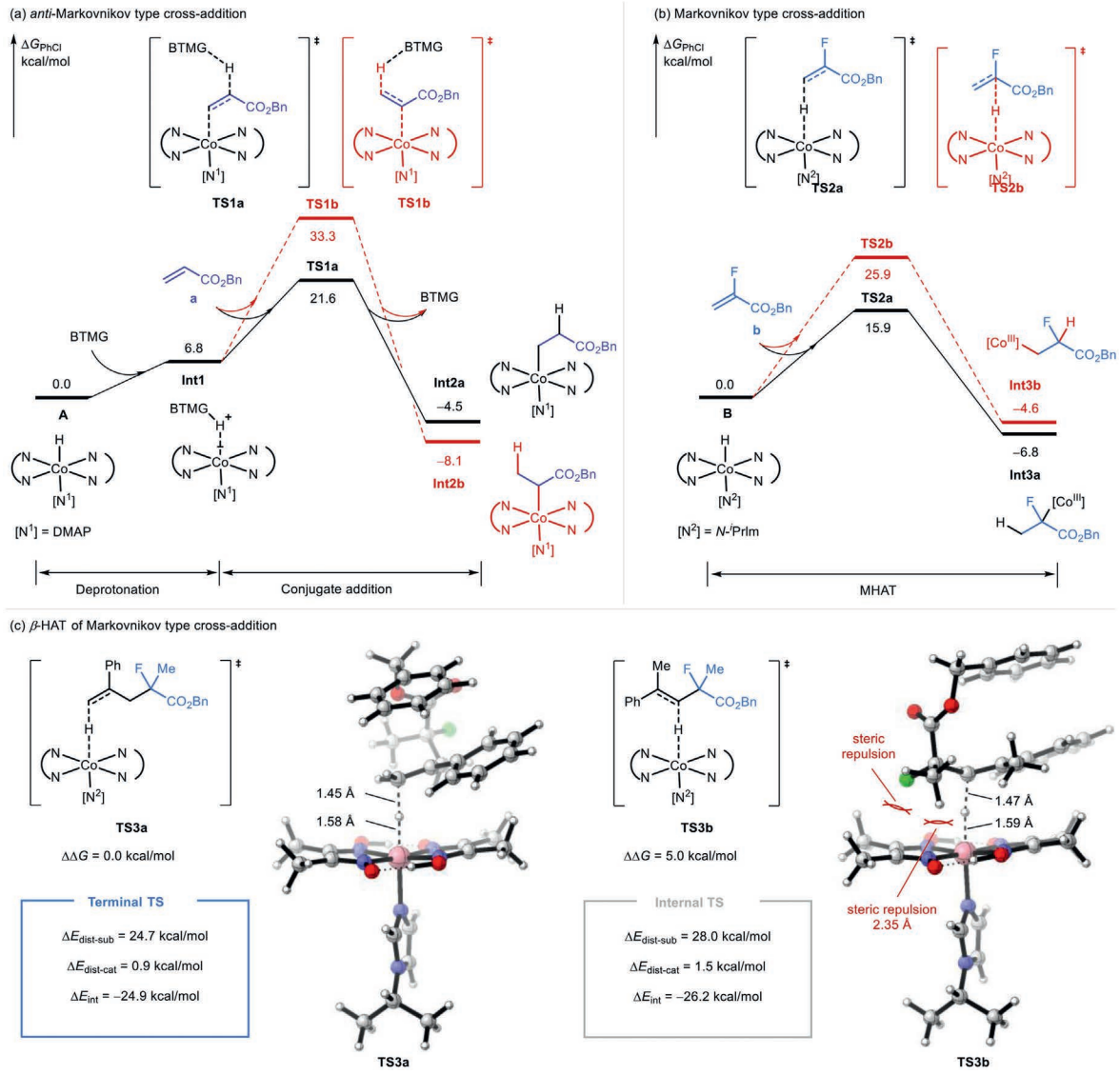
We next conduct a series controlling experiment to gain insight into the reaction mechanism. Deuterium labelling experiments demonstrated that the proton in the cross-addition products was from styrene (Scheme 5a). For the *anti*-Markovnikov hydroalkenylation of alkenes, the deuteration ratio of **8a** was 62 % (D_b) when 20 mol% of Co(dmgH)$_2$(DMAP)^{n}Bu was added. When catalyst loading was reduced to 10 mol%, the deuteration ratio of **8a** was 71 %. This indicates that the Co(dmgH)$_2$(DMAP)^{n}Bu can produce Co-H species *in situ* under light irradiation to initiate the catalytic cycle, and the H on cobalt can be incorperated into the product formation [40]. Identical conclusion can be deduced for Markovnikov hydroalkenylation reaction with the formation of **8b** (see Page S97 in Supporting information). In addition, the proposed Co-H was trapped by the triphenylmethyl cation to give product **8c** under blue light irradiation, while **8c** was not observed under dark (Scheme 5b). When TEMPO (1.0 equiv.) was added, the hydroalkenylation reaction was completely inhibited, resulting in the formation of TEMPO-trapped adducts **8d** and **8e**, showing that radical intermediate is likely involved in the transformation (Scheme 5c). Furthermore, by integrating insights from the litera-



Scheme 4. Markovnikov-selective hydroalkenylation of 2-substituted acrylates with α-methylstyrene. Standard conditions: 0.20 mmol scale, **1′**:**2′** = 3:1, $Co(dmgH)_2$(3,4-dimethylbenzylamine)Cl (10 mol%), Zn (1.0 equiv.), dioxane (c = 0.2 mol/L) at 75 °C for 18 h under Ar; [a] $Co(dmgH)_2$(Propylamine)Cl; [b] 0.20 mmol scale, **1′**:**2′** = 5:1.



Scheme 5. Control experiments.



Scheme 6. DFT studies and computed free energy profiles. (a) *anti*-Markovnikov type cross-addition catalyzed by Co(dmgH)$_2$(DMAP)H (A). (b) Markovnikov type cross-addition catalyzed by Co(dmgH)$_2$(*N*-^{i}PrIm)H (B). (c) Comparison of β-HAT transition states determining the regioselectivity. All energies were computed at the SMD/(U)B3LYP-D3(BJ)/def2-TZVP//(U)B3LYP-D3(BJ)/SDD-6–31G(d,p) level of theory.

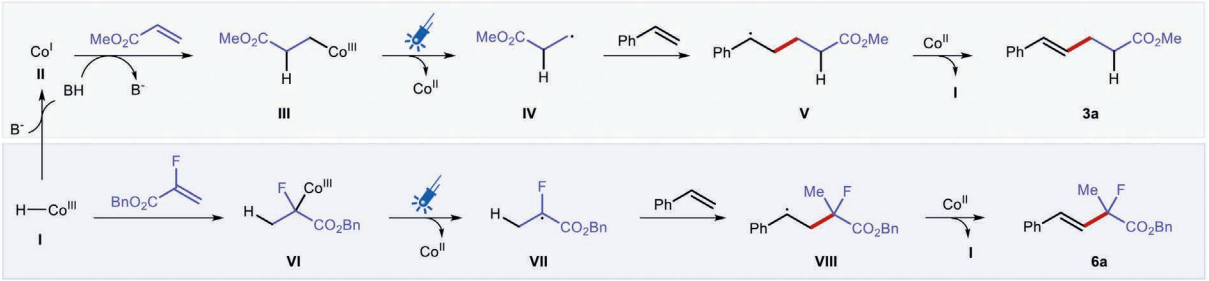
ture [41–47] with deuterium–labeling and radical–trapping experiments, we have demonstrated that the reaction does not proceed *via* reduction of the electron–deficient alkene to a radical–anion intermediate (see Page S103 in Supporting information). Light on/off experiment can exclude the possibility of a radical chain process (Scheme 5d).

We then performed DFT studies to further elucidate the reaction mechanism. For the *anti*-Markovnikov type cross-addition catalyzed by Co(dmgH)$_2$(DMAP)H (Scheme 6a), the Co^{III}-H species (**A**) first undergoes BTMG-promoted deprotonation to generate a nucleophilic Co^{I} intermediate (**Int1**). Subsequent conjugate addition with substrate a occurs *via* **TS1a** ($\Delta G^{\neq} = 21.6$ kcal/mol) to give **Int2a**, whereas the competing pathway through **TS1b** requires a higher barrier ($\Delta G^{\neq} = 33.3$ kcal/mol) to afford **Int2b**. These results align well with the experimentally observed regioselectivity (Scheme 2). The ensuing alkene insertion followed by β-hydrogen atom transfer (β-HAT) from **Int2a** delivers the *anti*-Markovnikov product with an overall barrier of 26.8 kcal/mol (Fig. S2 in Supporting information).

In the Markovnikov type cross-addition catalyzed by Co(dmgH)$_2$(*N*-^{i}PrIm)H (Scheme 6b), the Co^{III}-H species (**B**) undergoes Markovnikov-selective MHAT process with substrate **b** *via* **TS2a** ($\Delta G^{\neq} = 15.9$ kcal/mol) to form **Int3a**. The alternative *anti*-Markovnikov MHAT through **TS2b** is energetically less favorable ($\Delta G^{\neq} = 25.9$ kcal/mol), consistent with the radical-trapping experiments (Scheme 5c). Subsequent alkene insertion and β-HAT from **Int3a** afford the Markovnikov cross-addition product with an activation energy barrier of 21.7 kcal/mol (Fig. S3 in Supporting information).

We further investigated the β-HAT regioselectivity in the Markovnikov pathway (Scheme 6c). The transition state leading to the terminal alkene (**TS3a**) is 5.0 kcal/mol lower in energy than that forming the internal alkene (**TS3b**), matching the experimental product distribution (Scheme 4). In **TS3b**, steric repulsion between the substrate's methyl and fluorine groups and the Co-ligand framework raises the energy, accounting for the observed selectivity. Distortion/interaction analysis indicates that substrate distortion ($\Delta E^{\neq}_{\text{dist-sub}}$) is the dominant factor [48,49], highlighting the crucial role of steric effects in governing the β-HAT step.

Based on the results of the mechanistic studies and literature knowledge [40,50], we proposed that the catalytic cycle initiates with the light induced generation of Co^{III}-H species **I**, which has



Scheme 7. Proposed mechanism.

a pK_a about 10 and can be deprotonated by BTMG to afford the nucleophilic Co^I intermediate **II** (Scheme 7) [51]. Conjugate addition of Co^I to electron-deficient alkenes occurs, resulting in the formation of a linear alkylcobalt species **III** [52]. Upon light-induced homolytic cleavage of the Co-C bond of **III**, a primary alkyl radical **IV** is generated with the formation of Co^{II} intermediate [47,53]. Through radical addtion of **IV** to stryene, a new benzylic radical **V** formed. Finally, **V** undergoes β-H abstraction by Co^{II} to form *anti*-Markovnikov type product **3a**, with the generation of Co^{III}-H simultaneously to close the catalytic cycle [54–57]. In the absence of a base, Co^{III}-H (**I**) undergoes selective hydrocobaltation with benzyl 2-fluoroacrylate, forming the branched alkylcobalt species **VI**. As mentioned before, due to its strong electron-withdrawing effect, the MHAT [58–62] process was promoted by the incorporation of fluorine atom, which can also stabilize the resulting radical intermediate **VII**. This intermediate then engages in radical addition with styrene, followed by β-H abstraction, to afford the Markovnikov type product **6a** and regenerate **I**.

In summary, a cobalt-catalyzed system for the regiodivergent hydroalkenylation of electron-deficient alkenes with styrenes has been established. Under different oxidation state, the cobalt complexes displayed different reacitivity, to activate electron-deficient alkenes both in Markovnikov and *anti*-Markovnikov manner, thus to successfully realize regiodivergent C–C bond formation from terminal alkenes. Various (hetero)aryl alkenes and electron-deficient alkenes can be converted smoothly, with excellent regioselectivity, chemoselectivity and functional group tolerance. Mechanistic insights from DFT studies further rationalize the experimental observations and clarify the origin of the observed regioselectivities.

CRediT authorship contribution statement

Jianyi Shi: Writing – original draft, Methodology, Investigation, Data curation. **Chengyi Peng:** Investigation, Data curation. **Jiali Qiu:** Data curation. **Shuanglong Li:** Data curation. **Wenyan Zhao:** Investigation. **Xiang Sun:** Writing – original draft. **Shikai Xiang:** Writing – original draft, Investigation. **Yandong Wu:** Methodology, Investigation. **Wenhan Xu:** Methodology, Data curation. **Fei Pan:** Writing – original draft, Project administration, Funding acquisition. **Linxing Zhang:** Writing – review & editing, Project administration, Methodology, Data curation. **Fei Ye:** Writing – review & editing, Resources, Project administration, Funding acquisition.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.cclet.2026.112824.

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